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# Concentrations of Ethoprop in the Soil and Runoff Water of a Small Agricultural Watershed



U.S. Department of Agriculture  
Science and Education Administration  
Agricultural Research Results • ARR-S-2/October 1979

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This publication is available from the Southeast Watershed Research Program, Science and Education Administration, P.O. Box 946, Tifton, Ga. 31794.

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Science and Education Administration, Agricultural Research Results, Southern Series, No. 2, October 1979.

Published by Agricultural Research (Southern Region), Science and Education Administration, U.S. Department of Agriculture, P.O. Box 53326, New Orleans, La. 70153.

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# Concentrations of Ethoprop in Soil and Runoff Water of a Small Agricultural Watershed

By Wilmer A. Rohde,<sup>1</sup> Loris E. Asmussen,<sup>2</sup> Ellis W. Hauser,<sup>3</sup> and Alva W. Johnson<sup>4</sup>

## ABSTRACT

The dissipation in soil and movement in runoff water of the [nematicide] ethoprop (O-ethyl S,S-dipropyl phosphorodithioate) from Cowarts sandy loam was studied in 1974 and 1975 to determine if runoff of this widely used chemical causes the [pollution of streams and downstream areas]. The studies were in a small agricultural watershed near Tifton, Ga., that was instrumented to measure surface and subsurface flow. Significant differences occurred between years in both decomposition of ethoprop in the soil and its movement in the runoff water. Most of these differences are attributed to the formulation of ethoprop used. In 1974, 2 days after injection of 6L liquid ethoprop at a depth of 15 cm at the rate of 9 kg active ingredient/ha, the soil contained concentrations of up to 710 ng/g in the top 10 cm, but after 30 days 93% decomposition had occurred. In 1975, 2 days after incorporation of 10G granular ethoprop into the soil to a depth of 15 cm at the same rate, the ethoprop concentration was 1,207 ng/g in the top 10 cm, and these higher concentrations were also found at lower depths than when the liquid formulation was used. Conversely, higher concentrations of ethoprop from the liquid formulation were found in runoff water in 1974 than from the granular formulation in 1975. Runoff water 2 or 3 days after applications of the liquid ethoprop and the granular ethoprop contained 283  $\mu\text{g/l}$  and 45  $\mu\text{g/l}$  of ethoprop, respectively. Furthermore, the movement of ethoprop in runoff water during 1974, stated as a percentage of the total amount applied, was 0.1% in surface runoff and 0.01% in subsurface runoff. From the granular formulation, 0.01% of the ethoprop moved in the surface water, but no subsurface flow occurred. Forced runoff from small plots produced ethoprop losses of 0.42% from the liquid formulation and 0.02% from the granular formulation. These data may be typical of small plots, but differences of this magnitude would not be expected in fields or watersheds following natural rainfall events. These data indicate that no

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hazard to the environment exists as a result of movement of ethoprop in surface or subsurface water. In excess of 90% of the ethoprop from either the liquid or granular formulation was decomposed in the soil. Index terms: ethoprop, nematicides, pesticide degradation, pesticide dissipation, pesticide persistence, pesticides, runoff, soil leaching, Southern Coastal Plain, water pollution, water quality, watersheds.

## INTRODUCTION

Nematodes cause economically important and often dramatic reduction in yields of field and horticultural crops, especially in sandy soils of the Southern Coastal Plain, and ethoprop (*O*-ethyl *S,S*-dipropyl phosphorodithioate) is widely used for their control. It is classified in toxicity category I with a dermal LD<sub>50</sub> of 26 mg/kg (Harris 1975). Previous studies have indicated that it is not persistent in soil and that its residual life varies with such factors as application rate, formulation applied (liquid or granular), organic content of soil, soil type, temperature, moisture, and microflora. In field tests, half-life varied from 3 to 35 days (Goring 1967, Huvor 1969). A laboratory percolation study (Huvor 1969) on a sandy loam indicated downward movement of 30 cm after application of 12.5 cm of water. Loss from volatilization is considered to be insignificant; at 26° C its vapor pressure is only  $3.5 \times 10^{-4}$  torr. In tests with 10G granular formulation, very little volatilized from dry granules and none was detected from wet granules, but large amounts of degradation products such as mercaptans and sulfides were produced (Huvor 1969). Menzer et al. (1971) found that corn and soybeans metabolized ethoprop to ethyl propyl sulfide, ethyl propyl sulfoxide, ethyl propyl sulfone, propyl disulfide, and phosphoric acids and their salts. Brodie (1971) conducted a study on the vertical movement of ethoprop in Tifton sandy loam by measuring the degree of control of root-knot nematode at various depths, and reported 90% control down to 20 cm after incorporation of ethoprop in the top 5 cm of soil. Harris and Hitchon (1969), in a study of effectiveness against soil insects, found ethoprop to be 100 times more effective against crickets in moist than in dry soil. Its activity-persistence curve indicated a half-life of about 4 weeks, with little activity after 8 weeks.

No research has measured the movement of ethoprop or any nematicide in runoff water from fields. Concern about potential pollution of streams and downstream areas prompted us to

study its loss and movement in surface and subsurface runoff water and its dissipation and downward movement in a Southern Coastal Plain soil. The study period was from January 1974 to January 1976. The 6L liquid (2.7 kg active ingredient/3.8 l) and 10G granular formulations of ethoprop were used in 1974 and 1975, respectively. Analogous studies concerning the movement of 2,4-D in surface and subsurface runoff water reported by White et al. (1976) served as a model for our study.

The watershed-runoff studies were accompanied by supporting studies of forced runoff. By artificially applying water to small plots within the watershed, rates of ethoprop loss at selected intervals could be established. The decomposition curve for these simulated events supplemented the natural runoff events and permitted the development of decomposition curves representative of ethoprop decomposition for the entire year.

## MATERIALS AND METHODS

The study area, a 0.34-ha watershed called station Z, is located near Tifton, Ga., on Cowarts loamy sand containing 0.05% organic matter. The surface soil is underlain at depths of 92 to 214 cm by material having low water permeability; hence, downward movement of water is impeded, and water and dissolved chemicals mostly move from the area by lateral flow. This area has been described in detail by Jackson et al. (1973).

Rainfall in 1974 and 1975 was 124.2 and 131.4 cm, respectively (the 50-year average is 116.3 cm), and rainfall distribution during 1974 and 1975 was similar to the long-term monthly average. Hence, the study period was representative of the long-term precipitation patterns.

The watershed was instrumented for measuring surface and subsurface waterflow. Surface runoff water was collected in a grassed waterway adjoining the field and directed through a 30.5-cm H-flume. Stages were monitored continuously by an analog stage recorder. Subsurface flow was



collected by a tile drain along the lower edge of the drainage area. Tile depth ranged from 61 to 153 cm, depending on the topography. Waterflow from the area was directed through a 90° V-notch weir. A binary digital recorder monitored subsurface stages at 5-min intervals. A digital rain gage on the watershed boundary monitored rainfall by 5-min periods to 0.25 cm.

During 1974 and 1975 a winter cover crop of rye, *Secale cereale* L. cv. 'Wrens', was planted on the watershed. Each year the cover crop was cut and removed; the land was turned, disked, and bedded; 560 kg/ha of 0-10-20 fertilizer were applied; and 53.8 kg/ha of soybeans were planted. Other chemicals applied were 1.12 kg active ingredient (a.i.)/ha of trifluralin<sup>5</sup> (one time each season) and 0.5 kg a.i./ha of methomyl<sup>6</sup> (three times each season). The soybeans were harvested November 13, 1974, and November 18, 1975. In 1974 6L liquid ethoprop was applied on July 10 to the watershed by injection at a depth of about 15 cm at the rate of 9 kg a.i./ha. In 1975 10G granular ethoprop was applied at the same rate on May 12 and incorporated into the soil to a depth of 15 cm with a Rototiller.

Although ethoprop was not applied until July 10, 1974, water samples (250-ml) were collected beginning January 1 and analyzed for background and interfering organic compounds. Samples taken shortly after ethoprop application were collected with increasing frequency on the rising side of the hydrograph and with decreasing frequency on the receding side. Sampling frequency in the late summer and fall decreased. Composite samples were collected during the winter to determine the presence of ethoprop. Sample collection continued through 1975. Samples were refrigerated at about -15°C immediately after collection until analysis.

In 1974 simulated rainfall was applied to 28-m<sup>2</sup> subplots on July 12, August 9, and September 20—2, 30, and 72 days after ethoprop application. In 1975 the artificial runoff tests were conducted on May 23, June 3, and June 20—11, 22, and 39 days after ethoprop application. A natural rainfall on May 16, 4 days after ethoprop application, replaced a scheduled initial simulated rain.

The applicator for simulated rainfall was made

from two 2.5-cm pipes 8.23 m long, with six nozzles spaced every 2.5 m, each producing a square water pattern. The pipes were mounted 1.5 m above the ground. The area on which the water was applied included two beds with two rows of soybeans per bed. Water from deep wells was used for the water supply, and a pressure gage was used to control the application rate. Simulated rainfall was applied at 19.1 cm/h for approximately 30 min. The simulator was moved to adjacent beds after every test. Runoff water was collected in a gutter at the edge of the plot and directed through a small H-flume for measurement and sample collection.

Soil samples were collected with a 3- by 30-cm bucket-type auger. Sampling sites were randomly selected in the watershed at 12 locations for each sample date. Depths sampled in 1974 were 0-10, 10-20, and 20-60 cm, and in 1975, 0-10, 10-20, 20-30, and 30-60 cm. Sampling dates in 1974 were July 12 and 24, and August 9. In 1975, they were May 12, 14, 16, 19, 23, 27, and 31; June 6 and 13; July 9; and September 3. Sample sites selected were on the beds between the soybean rows. Soil samples were composited and mixed in a V-blender. Representative samples were then taken from the composite for ethoprop analysis, and the remaining samples were stored at -15°C. Samples usually were processed within 2 weeks after collection. Fortified check samples were prepared and stored with the field samples.

The sediment in runoff samples was thoroughly suspended before a 100-ml sample for ethoprop analysis was withdrawn. The sample was placed in a 300-ml french square bottle with 50 ml of pesticide-grade benzene and extracted for 30 s, using a Polytron ultrasonic homogenizer. The water layer was separated in a separatory funnel and reextracted with an additional 50 ml of benzene. The combined organic extract, was washed with saturated NaCl solution, 3x25 ml (3 times with 25 ml), and dried with Na<sub>2</sub>SO<sub>4</sub> before evaporating to dryness. The residue was taken up in hexane and diluted to 10 ml for analysis. This extraction procedure resulted in more than 98% recovery with no interfering peaks, as determined with fortified check samples. An analysis of four samples was done on the sediment in the water and on the filtered water. The filtered sample contained ethoprop concentrations of 50, 46, 161, and 170 µg/l and the sediment sample 50, 38, 282, and 171 µg/l. There was practically no adsorbance of the pesticide on the soil particles.

<sup>5</sup>A herbicide, *a,a,a*-trifluoro-2,6-dinitro-*N,N*-dipropyl-*p*-toluidine.

<sup>6</sup>An insecticide and nematicide, *S*-methyl *N*-[(methylcarbamoyl)oxy]thioacetimidate.

Table 1. — Rainfall, water discharge, and ethoprop loss from surface runoff, 1974 and 1975.

Month <sup>1</sup>	Rainfall (cm)	No. runoff events	Water discharge (area-cm)	Avg. ethoprop in samples (µg/l)	Ethoprop loss (g/ha)
<b>1974</b>					
1	11.18	3	0.538	0	0
2	15.16	4	1.407	0	0
3	8.74	...	.....	0	.....
4	11.79	3	.338	0	0
5	7.67	2	.183	0	0
6	15.16	...	.....	0	.....
7	15.37	4	1.986	42.3	8.392
8	10.59	1	.178	6.3	.112
9	17.35	4	.572	5.9	.336
10	1.63	...	.....	0	.....
11	2.54	...	.....	0	.....
12	7.06	...	.....	0	.....
<b>Total</b>	<b>124.24</b>	<b>21</b>	<b>5.202</b>	<sup>2</sup> 17	<sup>3</sup> 8.840
<b>1975</b>					
1	19.13	6	1.561	1.0	0.157
2	6.76	4	.542	1.0	.054
3	16.97	5	1.322	1.0	.020
4	24.41	5	1.316	1.0	.129
5	5.44	3	.191	50.8	.937
6	5.33	...	.....	.....	.....
7	10.44	2	.887	.18	.016
8	13.39	5	.382	.1	.004
9	10.06	...	.....	.....	.....
10	6.02	...	.....	.....	.....
11	3.07	...	.....	.....	.....
12	10.52	...	.....	.....	.....
<b>Total</b>	<b>131.54</b>	<b>30</b>	<b>6.201</b>	<sup>2</sup> 2.1	<sup>4</sup> 1.317

<sup>1</sup>After ethoprop application: July 10, 1974, and May 12, 1975.<sup>2</sup>Average of all samples.<sup>3</sup>Ethoprop lost: 0.1% of 9-kg/ha application of liquid formulation (6L).<sup>4</sup>Ethoprop lost: 0.015% of 9-kg/ha application of granular formulation (10G).

Soil samples were removed from the freezer as needed for analysis and airdried to less than 0.5% moisture; 100-g samples were extracted in a Waring Blendor with benzene (2×100 ml) for 5 min. The benzene extract was filtered through a Buchner funnel, and the filter cake was washed with an additional 50 ml of the solvent. The filtrate was transferred to a separatory funnel and washed with saturated NaCl solution (3×25 ml) and dried by filtering through Na<sub>2</sub>SO<sub>4</sub>. Further preparation corresponded to that used with the water samples. Recovery rates as determined from fortified check samples in the range of 1,000, 250, and 25 ng/g were more than 95%. These fortified sample extracts were used as calibration standards for gas-liquid chromatography.

Analysis for ethoprop was done on a Hewlett-Packard 5750 gas chromatograph fitted with a nitrogen/phosphorus thermionic detector. A glass column (6 mm by 180 cm) packed with 3% Poly S-179 on 100/120 mesh Gas-Chrom Q was used; carrier gas was helium at 50 ml/min. Inlet, oven, and detector temperatures were 220°, 220°, and 300° C, respectively. Triplicate 2-µl on-column injections were made from each sample vial. Results were recorded with a Hewlett Packard 3380 reporting integrator calibrated in the external standard mode. The average of the three determinations is reported. The standard deviations varied from 0.001 for 5-ng/g concentration to 0.009 for 1,000 ng/g (1 part per million). Response over this range of ethoprop

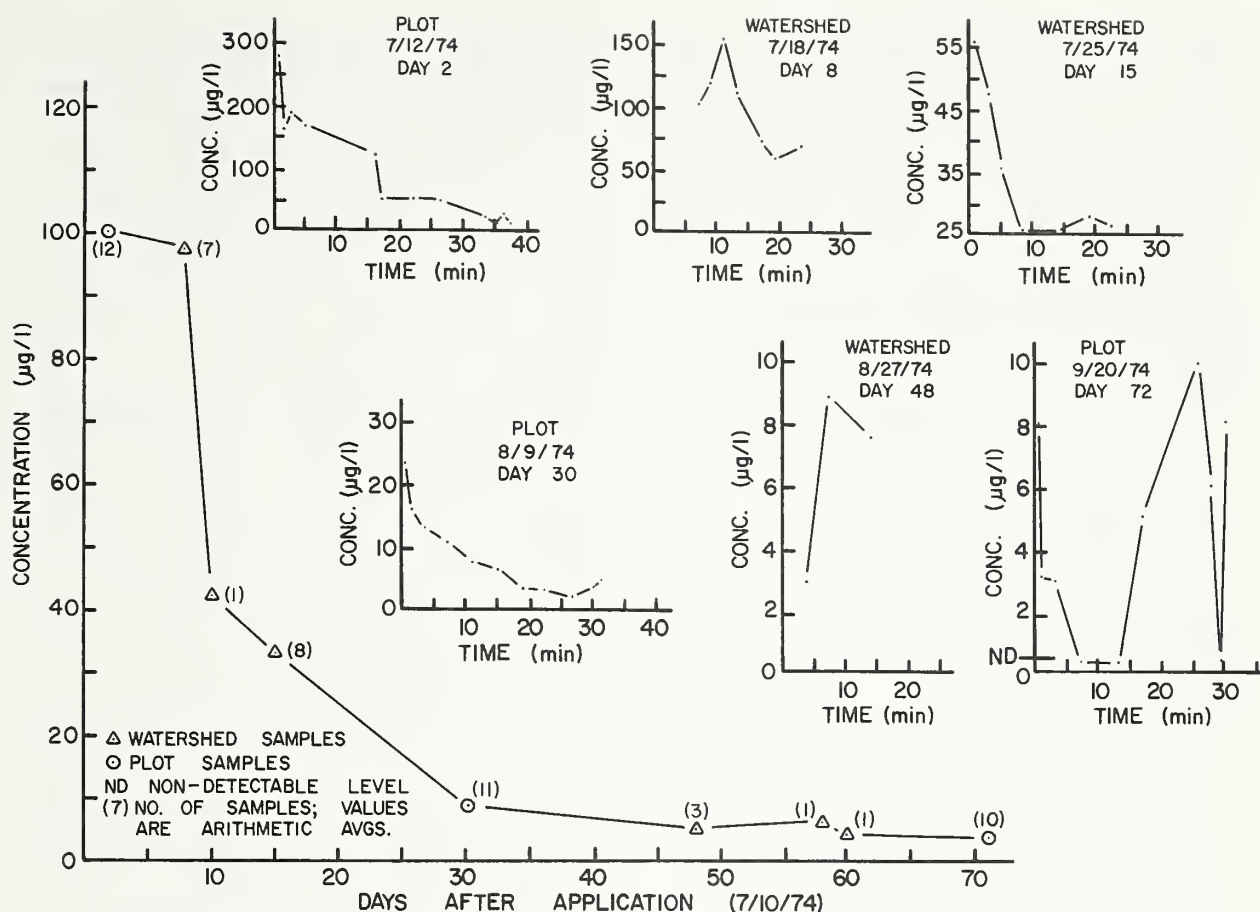


FIGURE 1.—Concentrations of ethoprop in natural watershed runoff and in artificial plot runoff, 1974.

concentration was linear for this detector and column.

## RESULTS AND DISCUSSION

### RUNOFF ANALYSES

Table 1 gives the monthly rainfall, water discharge, and average ethoprop concentrations in surface runoff from the watershed for 1974 and 1975. Ethoprop movement in subsurface water occurred only in 1974; these data are given in table 2. The first rainfall and natural runoff occurred 8 days after application of liquid ethoprop in 1974. A maximum concentration of  $153.6 \mu\text{g/l}$  of the nematocide was found during this event (7/18/74); this peak occurred after an initial concentration (first sample) of  $101.6 \mu\text{g/l}$ . The concentration dropped to  $71.6 \mu\text{g/l}$  in the seventh and last sample of this event. This information

and the data for all individual samples taken during events occurring in the 1974 study are shown in figure 1.

The data for the forced runoff events on the small plots are presented in table 3. During the first event, 12 samples were collected over the 37-min runoff period. The maximum concentration of  $283 \mu\text{g/l}$  was found in the initial sample (fig. 1); we found no slight "induction" period—a rise to a maximum in the second or third sample taken—as we generally found in the runoff samples from the watershed during natural events. This induction period is possibly explained by the delayed runoff during a natural rainfall. In figure 1 the concentration, or sample decomposition, curves for artificial plot runoff can be compared with those for natural watershed runoff during the 72-day season. The subsequent weighted average concentrations for events, natural and simulated, fall on a fairly smooth curve approximating a first-order decomposition. Watershed sampling was



determined by the intensity and duration of the rainfall. For example, single samples were collected on the 10th, 58th, and 60th days, whereas three samples were obtained on the 48th day after chemical application. Even so, when the arithmetic averages are plotted with the single-sample analyses, a well-defined smooth concentration curve is obtained.

On May 15, 1975, 3 days after application of granular ethoprop, natural rainfall caused runoff with an ethoprop concentration of 45  $\mu\text{g/l}$  (fig. 2). During rainfall the following day, seven 1-l samples were collected within 40 min. Ethoprop

concentrations ranged from 26  $\mu\text{g/l}$  in the first sample to 42  $\mu\text{g/l}$  in the last. This is shown in detail in figure 2, where it is plotted on the event curve by sample concentration vs. time and on the seasonal curve with the arithmetic average concentration of 36  $\mu\text{g/l}$ . These first samples contained the maximum concentration measured for the entire year, including the simulated rainfall plot runoff of May 23. These findings contrast with the 1974 maximum concentration of 283  $\mu\text{g/l}$ , when the nematocide was applied as a liquid. The fact that this high concentration in 1974 occurred in a simulated event only 2 days after application as opposed to 4 days after application in 1975 does not account for this great difference. The concentration decomposition curves for both 1974 and 1975 are presented in figure 3. These plots of the arithmetic average of the ethoprop concentrations (micrograms per liter) for each event in the runoff, from both plots and watershed, define the curve for granular and liquid formulations. The runoff during these first 4 days in 1975 accounted for a loss of 0.01% of the applied nematocide, while the total seasonal loss in runoff was 0.015% (table 1). In 1974, the subsurface flow accounted for a loss of 0.01% of the applied ethoprop (table 2), while the surface runoff accounted for 0.1% of that applied to the watershed. This contrast in results from the 2-year study is even greater when comparing the runoff loss data from the plots under simulated rainfall (tables 3 and 4). In 1974 there was a 0.42% loss and in 1975 only a 0.02% loss in the plot runoff.

The decomposition of ethoprop for individual plot events in 1974 and 1975 is shown on figure 4. The magnitude of the differences is significant. Also, in 1975 the concentration of ethoprop was approximately 2  $\mu\text{g/l}$  10 days after application,

Table 2.—Water discharge and ethoprop loss from subsurface runoff, 1974

Month <sup>1</sup>	No. runoff events	Water discharge (area-cm)	Avg. ethoprop in samples ( $\mu\text{g/l}$ )	Ethoprop loss (g/ha)
1	2	0.119	0	0
2	2	8.545	0	0
3	...	.....	...	.....
4	1	3.208	0	0
5	...	.....	...	.....
6	...	.....	...	.....
7	1	.467	0	0
8	1	1.633	4.2	.671
9	1	7.395	.3	.224
10	...	.....	...	.....
11	...	.....	...	.....
12	...	.....	...	.....
Total	6	21.367	2.42	3.895

<sup>1</sup> After ethoprop application July 10, 1974.

<sup>2</sup> Average of all samples.

<sup>3</sup> Ethoprop lost: 0.01% of 9-kg/ha application of liquid formulation (6L).

Table 3.—Ethoprop loss in surface runoff resulting from 19.1-cm/h simulated rainfall on plots 2, 30, and 72 days after ethoprop application, 1974<sup>1</sup>

Rainfall	No. samples	Water discharge (area-cm)	Ethoprop loss (g/ha)	Wtd. avg. ethoprop in samples ( $\mu\text{g/l}$ )	Range of ethoprop in samples ( $\mu\text{g/l}$ )
July 12 . . . .	12	3.391	34.0	100.6	19.6 to 283.0
Aug. 9 . . . .	11	2.075	1.2	6.1	3.0 to 23.2
Sept. 20 . . .	10	5.992	2.2	3.8	>3.0 to 8.0
Total . . .	33	11.458	<sup>2</sup> 37.4	.....	.....

<sup>1</sup> Ethoprop applied July 10, 1974.

<sup>2</sup> Ethoprop lost: 0.42% of 9-kg/ha application of liquid formulation (6L).

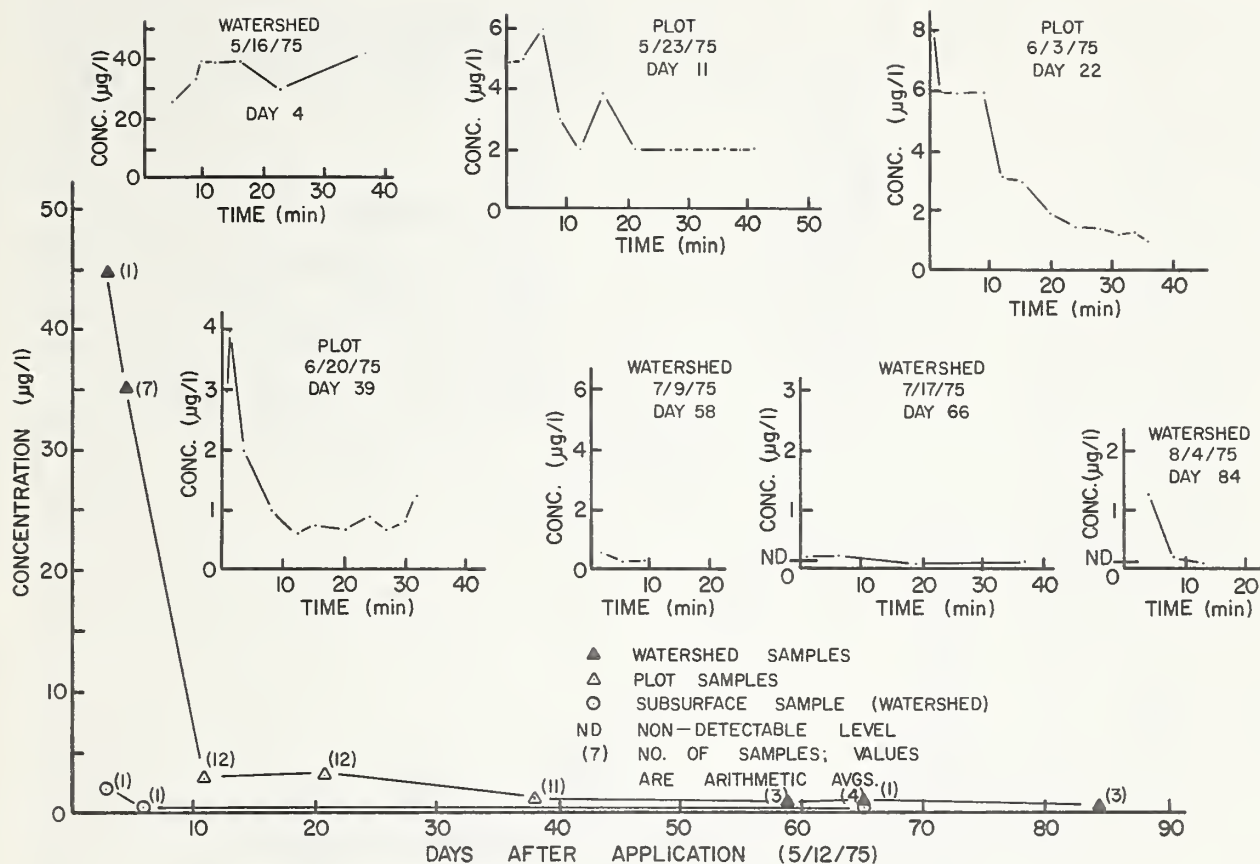


FIGURE 2.—Concentrations of ethoprop in natural watershed runoff and in artificial plot runoff, 1975.

Table 4.—Ethoprop loss in surface runoff resulting from 19.1-cm/h simulated rainfall on plots 11, 22, and 39 days after ethoprop application, 1975<sup>1</sup>

Rainfall	No. samples	Water discharge (area-cm)	Ethoprop loss (g/ha)	Wtd. avg. ethoprop in samples (µg/l)	Range of ethoprop in samples (µg/l)
May 23 ...	12	2.999	0.839	2.8	2.0 to 6
June 3 ....	12	2.777	.887	3.2	1.0 to 8
June 20 ...	11	3.143	.304	1.0	0.6 to 4
Total ...	35	8.918	<sup>2</sup> 2.029	...	.....

<sup>1</sup>Ethoprop applied May 12, 1975.

<sup>2</sup>Ethoprop lost: 0.02% of 9-kg/ha application of granular formulation (10G).

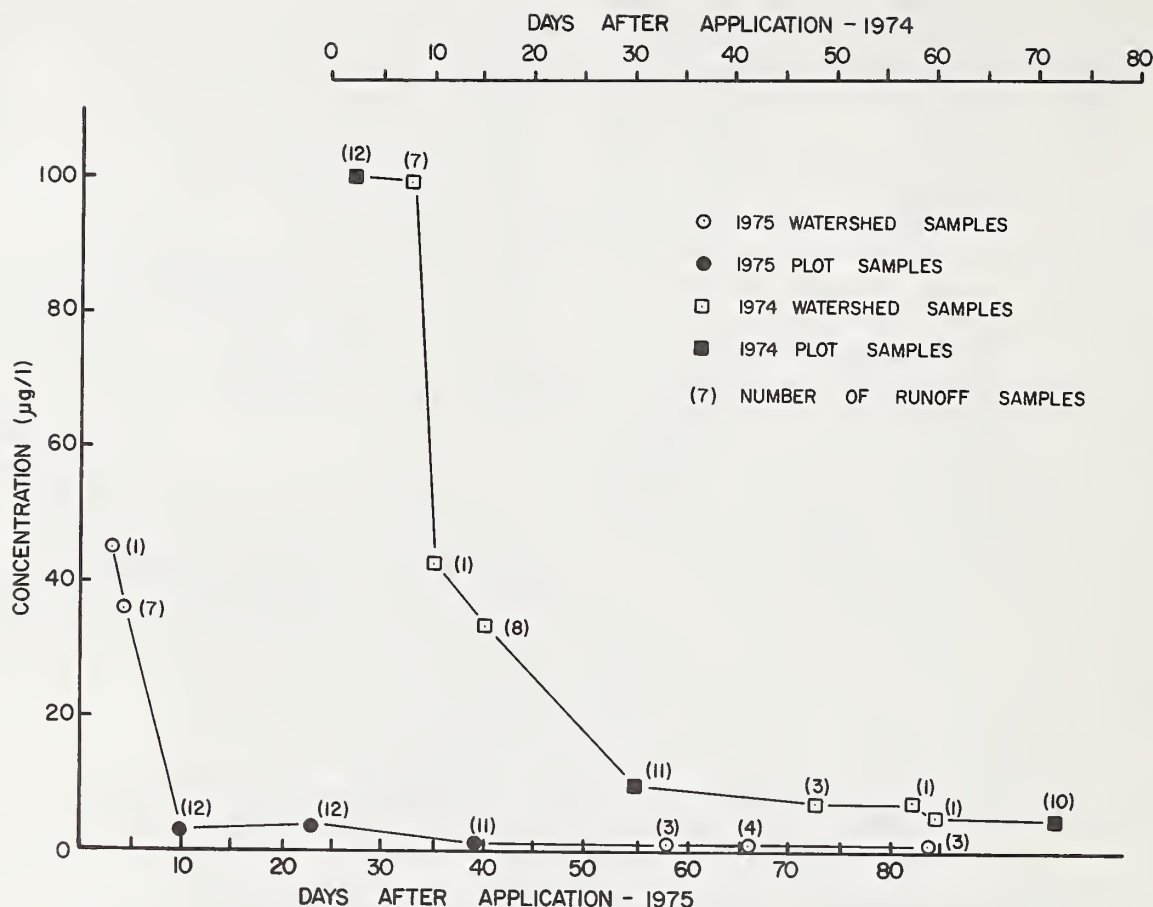


FIGURE 3.—Average concentrations of ethoprop in natural watershed runoff and in artificial plot runoff, 1974 and 1975.

while in 1974 it was approximately 10 µg/l 30 days after application. The potential for ethoprop loss when liquid application is made is obviously greater than it is for granular application. There was no subsurface runoff loss of ethoprop from the watershed in 1975. This also indicates that ethoprop in liquid formulation is more mobile and moves more readily in shallow phreatic groundwater flow.

#### SOIL ANALYSES

The dissipation of ethoprop in the soil was plotted in figure 5, which graphically illustrates the difference in amounts of ethoprop found in 1974 when the liquid formulation was applied and the amounts in 1975 when the granular formulation was applied. The concentration of ethoprop in 1974 was apparently unchanged during the first 2 weeks after application in the top 10 cm of soil, with a maximum concentration of 710 ng/g

of soil. The maximum concentration at 10-20 cm was 63 ng/g 2 days after incorporation; after 14 days there were only 26 ng/g. Thirty days after application residues had dropped to 52 ng/g of soil in the top 10 cm and to 12 ng/g at 10-20 cm. This rapid decline precluded later sampling. Ethoprop was not found in soil samples taken from below 20 cm.

In 1975, just before application of granular ethoprop, soil samples were collected from the watershed at depths of 0-10, 10-20, 20-30, and 30-60 cm. These samples contained ethoprop in the amounts of 20, 12, 3, and 1 ng/g of soil. These analyses were used as background values. The ethoprop concentrations in 1975, 2 days after application, were 1,207, 780, 120, and 129 ng/g of soil at the respective depths cited above. Soil samples collected from 0 to 10 cm 4 and 7 days after chemical application contained 886 and 692 ng/g of soil (57% of the second-day concentration), but the samples taken from 10 to 20



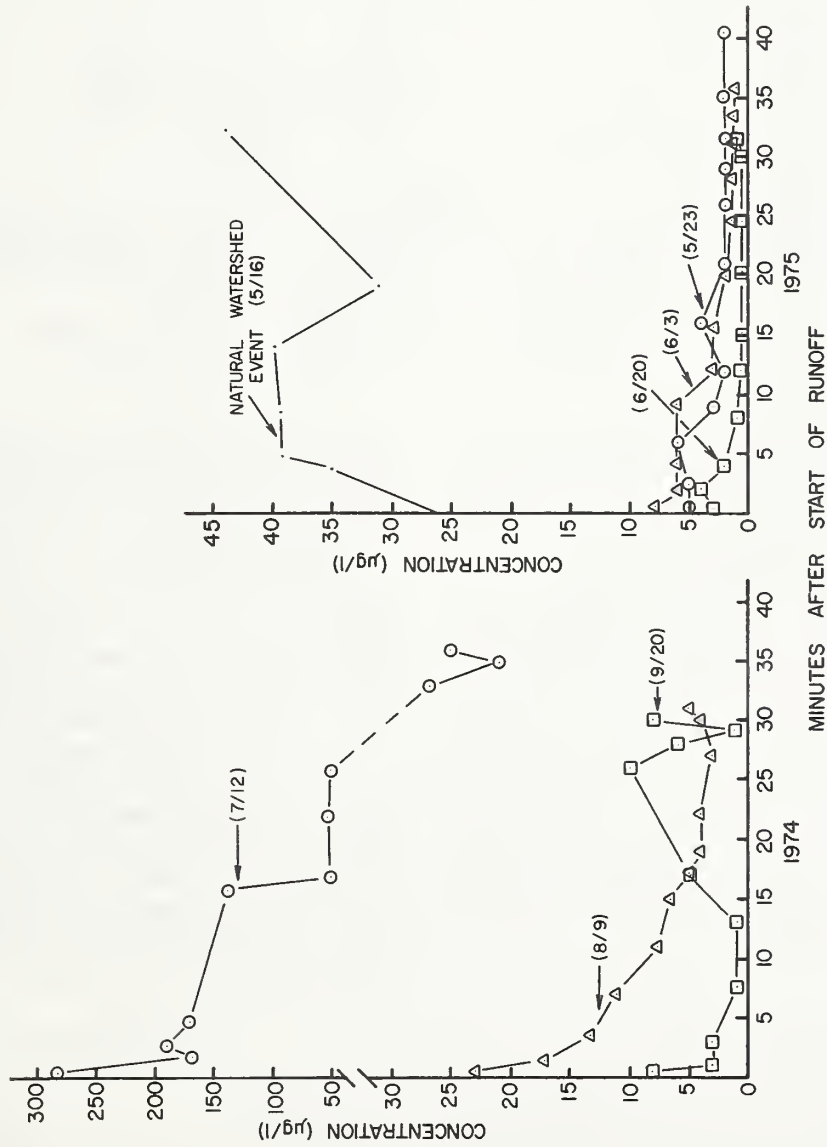


FIGURE 4.—Combined curves showing concentrations of ethoprop for all artificial runoff events in each year, 1974 and 1975.

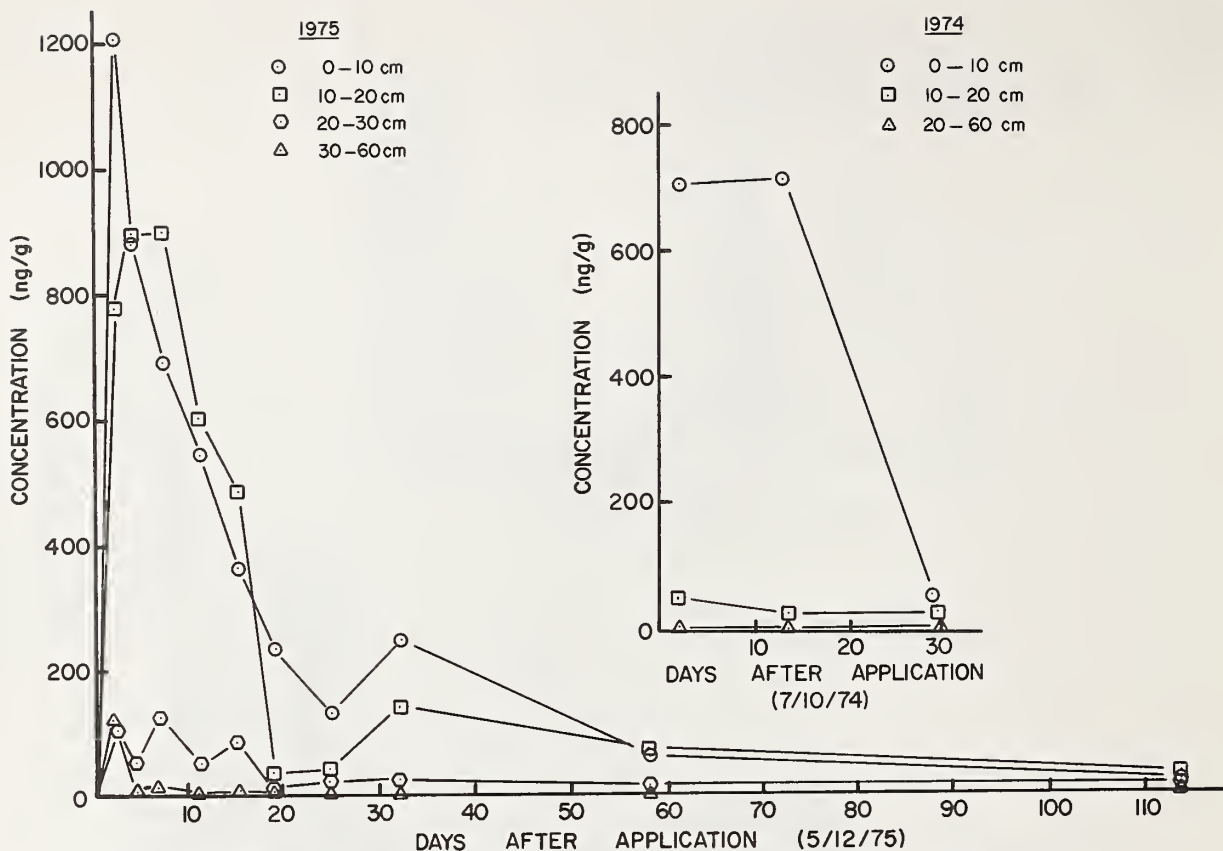


FIGURE 5.—Concentrations of ethoprop at various depths in the soil, 1974 and 1975.

cm contained 887 and 907 ng/g. Even the sample from 20- to 30-cm increased to 127 ng/g of soil after 7 days. Concentrations after 7 days decreased, approximating a first-order decomposition curve over the 114 days of sampling. The higher values shown at 32 days for the samples from 0-10 and 10-20 cm are likely because of nonuniform application of the granules. The lack of precision in uniform application of pesticides contributes to the inherent variation that occurs frequently in small soil samples from the field. With the use in laboratories today of highly automated processor-controlled analytical instruments having ultrasensitive, linear, element-selective detectors, the greatest error in precision and accuracy probably occurs in application and sampling of the pesticides.

## CONCLUSIONS

Significant differences were noted between the 1974 and 1975 ethoprop concentrations in runoff

from the watershed and in the decomposition of ethoprop in the soil. These differences probably can be attributed to the formulation of ethoprop used during the experiment—liquid in 1974 and granular in 1975. Total loss of the applied ethoprop during 1974 and 1975 in surface and subsurface runoff from the watershed did not constitute a significant loss, less than 0.1% in 1974 and 0.015% in 1975. Ethoprop concentrations in the runoff from the granular-formulation treatment was less than one-half that of the liquid. The persistence in the soil and downward movement of ethoprop was also assumed to be related to the formulation. Liquid ethoprop was found in the subsurface flow from the watershed. Granular ethoprop was persistent in the soil over a longer period of time and reached a higher initial concentration than liquid ethoprop.

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